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1/77

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1. Your reference — SMC 60659/GB/P1

2. Patent application number *(The Patent Office will fill in this part)* — 15 NOV 2002 0226708.6

18NOV02 E763922-1 D02944
P01/7700 0.00-0226708.6

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

Avecia Limited
Hexagon House
Blackley
Manchester, M9 8ZS
United Kingdom

Patents ADP number *(if you know it)* 07764137001

If the applicant is a corporate body, give the country/state of its incorporation: GB

4. Title of the invention

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5. Name of your agent *(if you have one)*

MAYALL, John

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

Avecia Limited
Hexagon House
Blackley
Manchester, M9 8ZS
United Kingdom

Patents ADP number *(if you know it)* 6244313002 06244313004

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application	Date of filing *(day / month / year)*

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*

a) *any applicant named in part 3 is not an inventor, or*
b) *there is an inventor who is not named as an applicant, or*
c) *any named applicant is a corporate body.*
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Continuation sheets of this form	
Description	18
Claim(s)	04
Abstract	
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10. If you are also filing any of the following, state how many against each item.

Priority documents	
Translations of priority documents	
Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	
Request for preliminary examination and search *(Patents Form 9/77)*	
Request for substantive examination *(Patents Form 10/77)*	
Any other documents *(Please specify)*	

11. I/We request the grant of a patent on the basis of this application.

Signature [signature] Date 15/11/2002

Avecia Limited Authorised Signatory

12. Name and daytime telephone number of person to contact in the United Kingdom: Mrs K.M. Pinder/Miss G. Terry 0161 721 1361/2

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Notes

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SMC 60559

APPLICANTS

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This invention relates to compounds, to processes for their preparation, to compositions derived therefrom and to their use in ink jet printing ("IJP"). IJP is a non-impact printing technique in which droplets of ink are ejected through a fine nozzle onto a substrate without bringing the nozzle into contact with the substrate.

There are many demanding performance requirements for dyes and inks used in IJP. For example they desirably provide sharp, non-feathered images having good water-fastness, light-fastness and optical density. The inks are often required to dry quickly when applied to a substrate to prevent smudging, but they should not form a crust over the tip of ink jet nozzles because this will stop the printer from working. The inks should also be stable to storage over time without decomposing or forming a precipitate which could block the fine nozzles.

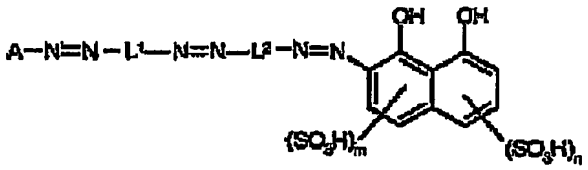
JP 58-174459 describes certain dis-azo dyes comprising once-coupled 1,8-dihydroxynaphthalene and their use in ink jet printing.

JP 57-36693 and US 4,395,288 describe certain tetra-azo dyes comprising 1,8-dihydroxynaphthalene and their use in ink jet printing.

US 1,209,154, published in 1916, describes the synthesis of certain tris-azo dyes comprising a 1,8-dihydroxynaphthalene group at one end and a 1,3-diaminobenzene group at the other end. These dyes are used for the conventional dyeing of cotton.

Surprisingly it has been found that the tris-azo dyes of the present invention have exceptional properties when used as ink jet inks, producing prints of high optical density (OD) of a neutral black with excellent light and ozone-fastness and high operability.

According to the present invention there is provided a process for printing an image on a substrate comprising applying thereto a composition comprising a liquid medium and a tris-azo compound of Formula (1) or salt thereof:



Formula (1)

wherein:

A is an optionally substituted alkenyl, a homocyclic or heterocyclic group;

L^1 and L^2 are each independently optionally substituted aryl or heteroaryl; and

m and n are each independently 0 or 1 such that m+n is 1 or 2;

wherein:

(i) the compound of Formula (1) is optionally in the form of a metal chelate; and

(ii) at least one of L^1 and L^2 carries at least one substituent selected from sulpho, carboxy, C_{1-4}-alkoxy and C_{1-4}-alkoxy-OH.

Preferably the composition is applied to the substrate by means of an ink jet printer. The ink jet printer preferably applies the composition to the substrate in the form of droplets which are ejected through a small orifice onto the substrate. Preferred ink jet printers are piezoelectric ink jet printers and thermal ink jet printers. In thermal ink jet printers, programmed pulses of heat are applied to the composition in a reservoir by means of a resistor adjacent to the orifice, thereby causing the composition to be ejected in the form of small droplets directed towards the substrate during relative movement between the substrate and the orifice. In piezoelectric ink jet printers the oscillation of a small crystal causes ejection of the composition from the orifice.

The image is preferably text, a picture, a photorealistic image or a combination thereof.

The substrate is preferably paper, plastic, metal or glass, more preferably a treated substrate such as a coated paper or coated plastic, especially plain paper. One of the advantages of the present process is its ability to provide very good printing results even on plain paper.

Preferred papers have an acid, alkaline or neutral character. Examples of commercially available treated papers include HP Premium Coated Paper™, HP Photopaper™, HP Printing paper ™ (available from Hewlett Packard Inc.); Stylus Pro 720 dpi Coated Paper™, Epson Photo Quality Glossy Film ™, Epson Photo Quality Glossy Paper ™ (all available from Seiko Epson Corp.); Canon HR 101 High Resolution Paper™, Canon GP 201 Glossy Paper™, Canon HG 101and HG201 High Gloss Film ™, Canon PR101 ™ (all available from Canon); Kodak Premium Photopaper, Kodak Premium InkJetpaper ™ (available from Kodak); Konica Inkjet Paper QP™ Professional Photo Glossy, Konica Inkjet Paper QP™ Professional Photo 2-sided Glossy, Konica Inkjet Paper QP™ Premium Photo Glossy, Konica Inkjet Paper QP™ Premium Photo Silky ™ (available from Konica) and Xerox Acid Paper (available from Xerox).

In this specification any groups shown in the free acid form also include the salt form. Furthermore the formulae shown in this specification cover all tautomers thereof.

When the compound of Formula (1) is in the form of a salt preferred salts are alkali metal salts, especially lithium, sodium and potassium salts, ammonium and substituted ammonium salts and mixtures thereof. Especially preferred salts are salts with ammonia and volatile amines. The free acid form may be converted into a salt using known techniques. For example, an alkali metal salt may be converted into a salt with ammonia or an amine by dissolving an alkali metal salt of the composition in water, acidifying with a mineral acid and adjusting the pH of the solution to pH 9 to 9.5 with ammonia or the amine and removing the alkali metal cations by dialysis.

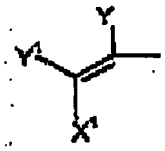
The preferred optionally substituted homocyclic or heterocyclic group groups represented by A are optionally substituted aryl, heteroaryl and non-aromatic cyclic groups.

Preferred optionally substituted aryl groups represented by A, L^1 and L^2 are each independently optionally substituted phenyl, biphenyl or naphthyl. In another embodiment of the present invention it is preferred that A is optionally substituted heteroaryl. Preferred optionally substituted heteroaryl groups represented by A, L^1 and L^2 are any heterocycle or substituted heterocycle comprising a 5- to 7- membered ring. Similarly preferred non-aromatic heterocyclic groups represented by A comprising a 5- to 7- membered ring, preferably comprising at least one double bond.

Examples of heteroaryl groups include pyridyl, furyl, thienyl, thiazolyl, isothiazolyl, imidazolyl, benzimidazolyl, pyrazinyl, pyrimidyl, quinolyl, isoquinolyl, benzofuryl, benzothienyl, pyrazolyl, indolyl, purinyl, isoxazolyl, oxazolyl, thiadiazolyl and furazanyl groups.

Examples of non-aromatic cyclic groups include pyridonyl, pyrazolonyl, piperidinyl, piperazinyl, pyrrolidinyl, morpholinyl, tetrahydrofuranyl, tetrahydrothiophenyl and tetrahydropyranyl, with pyridonyl being especially preferred.

Preferred optionally substituted alkenyl groups are of the Formula (2) and tautomers thereof:



Formula (2)

wherein:

Y is an electron withdrawing group;

Y^1 is H, alkyl, aryl, OR or $N(R)_2$ in which each R independently is H, optionally substituted alkyl or optionally substituted aryl; and

X^1 comprises at least one heteroatom selected from N, O and S.

Y is preferably selected from CN, CO_2H, CO_2R, $CON(R)_2$, COR and $-SO_2N(R)_2$ in which each R independently is as defined above. When R is optionally substituted alkyl it is preferably C_{1-6}-alkyl, more preferably C_{1-4}-alkyl. When R is optionally substituted aryl it is preferably phenyl or naphthyl, more preferably phenyl. When R is optionally substituted alkyl or aryl optional substituents are preferably selected from water solubilising groups, particularly SO_3H, SO_2NH_2, CO_2H or PO_3H_2 and salts thereof

When Y^1 is alkyl it is preferably C_{1-6}-alkyl, more preferably C_{1-4}-alkyl. When Y^1 is aryl it is preferably phenyl.

X^1 is preferably OR, CO_2R or NR in which R is as defined above.

More preferably Y is CO_2R^1, Y^1 is OR^1 and X^1 is OR^1 wherein each R^1 independently is H or C_{1-4}-alkyl.

Optional substituents which may be present on A , L^1 and L^2 are preferably selected from OH, SO_3H, CN, carbonamido, PO_3H_2, CO_2H, NO_2, NH_2, optionally substituted alkyl (especially C_{1-4}-alkyl optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group), optionally substituted alkoxy (especially C_{1-4}-alkoxy optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, C_{1-4}-alkyl, amino or hydroxy group), optionally substituted aryl (especially phenyl or phenyl carrying from 1 to 3 substiuents selected from sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino, hydroxy and N carrying one or two C_{1-4}-alkyl groups optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group), optionally substituted amine (especially N carrying one or two C_{1-4}-alkyl groups optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group) and optionally substituted acylamine (especially C_{1-4}-acylamino).

Preferably A carries from 0 to 5 substituents, more preferably 1 to 4 substituents, especially 1, 2 or 3 substituents. In a preferred embodiment A is not 1,3-diaminophenyl.

As examples of optionally substituted phenyl and naphthyl groups represented by A there may be mentioned 2,4-dihydroxyphenyl, 3-sulfo-4,6-diaminophenyl, 2-hydroxy-4-diethylaminophenyl 2-sulfo-4-diethylaminophenyl, 1-hydroxy-3,6-disulphonaphthyl and 1,8-dihydroxy-3,6-disulfonaphthyl. Preferred optionally substituted heteroaryl groups represented by A are any heterocycle or substituted heterocycle comprising a 5- to 7-membered ring, more preferably optionally substituted pyridyl, pyrazolyl or 1,2,4-triazolyl.

Preferably L^1 and L^2 are each independently, or comprise, one or more arylene groups, more preferably one or two optionally substituted phenylene or naphthylene groups. When L^1 or L^2 is, or comprises, more than one arylene group, the said arylene groups are optionally connected by means of a covalent bond or group containing from 1 to 10 atoms selected from O, S, N, C, H and combinations thereof, for example –O-, $-NR^2$-, $-NR^2$-CO-, $-NR^2CONR^2$-, -S-, -SO-, $-SO_2$-, $-SO_2NR^2$- or $-C\ R^2{=}C\ R^2$-, wherein each R^2 independently is H or C_{1-4}-alkyl.

Preferably L^1 and L^2 are each independently optionally substituted phenylene or naphthylene, wherein at least one of L^1 and L^2 carries at least one substituent selected from sulpho, carboxy, C_{1-4}-alkoxy and C_{1-4}-alkoxy-OH. Preferably L^1 and L^2 each independently carries from 0 to 3 substituents, more preferably 1 or 2 substituents, such that at least one of L^1 and L^2 carries at least one substituent selected from sulpho and carboxy. Further preferably at least one of L^1 and L^2 carries at least one substituent selected from C_{1-4}-alkoxy and C_{1-4}-alkoxy-OH

Preferably L^1 carries a substituent selected from sulpho and carboxy and L^2 carries at least one substituent selected from sulpho, carboxy C_{1-4}-alkoxy and C_{1-4}-alkoxy-OH;

As examples of optionally substituted phenylene and naphthylene groups represented by L^1 and L^2 there may be mentioned 2-sulphophenylene, 2-carboxyphenylene, 2,5-dihydroxyethyloxyphenylene and 7-sulphonaphthylene.

When the compound of Formula (1) is in the form of a metal chelate the metal is preferably Boron or a transition metal, more preferably Mn, Fe, Cr, Co, Ni, Cu or Zn, especially Co, Ni or Cu. The metal may be complexed with the compound of Formula (1) in a ratio of from 1:2 to 2:1, preferably in a ratio of metal to compound of Formula (1) of 1:2, 2:3, 1:1, 2:2 or 2:1, especially 2:1. However we have found that when the compound of Formula (1) is not in the form of a metal chelate the compound is still a valuable colorant for ink jet printing. Such unmetallised dyes are cheaper and easier to make than the corresponding metal chelates and they are more environmentally friendly due to the absence of, for example, transition metals.

Preferably the compound of Formula (1) is black.

Bearing in mind the above preferences, the compound of Formula (1) is preferably of the Formula (1) wherein:

A is optionally substituted pyridyl, furyl, thienyl, thiazolyl, isothiazolyl, imidazolyl, benzimidazolyl, pyrazinyl, pyrimidyl, quinolyl, isoquinolyl, benzofuryl, benzothienyl, pyrazolyl, indolyl, purinyl, isoxazolyl, oxazolyl, thiadiazolyl, furazanyl, pyridonyl, pyrazolonyl, piperidinyl, piperazinyl, pyrrolidinyl, morpholinyl, tetrahydrofuranyl, tetrahydrothiophenyl or tetrahydropyranyl;

L^1 phenyl or naphthyl optionally carrying a substituent selected from sulpho and carboxy;

L^2 is phenyl or naphthyl carrying at least one substituent selected from sulpho, carboxy, C_{1-4}-alkoxy and C_{1-4}-alkoxy-OH; and

m and n are each independently 0 or 1 such that m+n is 1 or 2;

wherein said optional substituents are selected from OH; SO_3H; CN; carbonamido; PO_3H_2; CO_2H; NO_2; NH_2; C_{1-4}-alkyl optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group; C_{1-4}-alkoxy optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, C_{1-4}-alkyl, amino or hydroxy group; phenyl or phenyl carrying from 1 to 3 substituents selected from sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino, hydroxy and N carrying one or two C_{1-4}-alkyl groups optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group; N carrying one or two C_{1-4}-alkyl groups optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group; and C_{1-4}-acylamino.

According to a second aspect of the present invention there is provided a tris-azo compound of Formula (1) or salt thereof as hereinbefore defined, with the provisos that (i) the compound of Formula (1) is optionally in the form of a metal chelate; (ii) at least one of L^1 and L^2 carries at least one substituent selected from sulpho, carboxy, C_{1-4}-alkoxy and C_{1-4}-alkoxy-OH; and (iii) when L^1 carries a methoxy group A is not 1,3-diaminophenyl.

The preferences for the compound according to the second aspect of the present invention are as described above in relation to the first aspect of the present invention, with

the proviso that when both groups represented by L are free from sulpho, carboxy and C_{1-4}-alkoxy-OH groups then A is not 1,3-diaminophenyl.

In a preferred group of compounds according to the invention at least one of the groups represented by L carries at least one group selected from

According to a third aspect of the present invention there is provided a composition comprising a tris-azo compound of Formula (1) or salt thereof as defined in relation to the first aspect of the present invention and a low melting point solid or a liquid medium comprising water and an organic solvent. Preferably the group represented by A in the compound of Formula (1) is not 1,3-diaminophenyl.

In one embodiment of the third aspect of the present invention the compound of Formula (1) is not of the formula shown in Example 8.

The composition preferably comprises:

(a) from 0.01 to 30 parts of a compound of Formula (1) or salt thereof as defined in relation to the first aspect of the present invention; and

(b) from 70 to 99.99 parts of a low melting point solid or a liquid medium comprising water and an organic solvent;

wherein all parts are by weight and the number of parts of (a)+(b)=100.

The process preferably uses the aforementioned composition.

The number of parts of component (a) is preferably from 0.1 to 20, more preferably from 0.5 to 15, and especially from 1 to 5 parts. The number of parts of component (b) is preferably from 99.9 to 80, more preferably from 99.5 to 85, especially from 99 to 95 parts.

Preferably component (a) is completely dissolved in component (b). Preferably component (a) has a solubility in component (b) at 20°C of at least 10%. This allows the preparation of liquid dye concentrates which may be used to prepare inks and reduces the chance of the dye precipitating if evaporation of the liquid medium occurs during storage.

The weight ratio of water to organic solvent is preferably from 99:1 to 1:99, more preferably from 99:1 to 50:50 and especially from 95:5 to 80:20.

It is preferred that the organic solvent present in the mixture of water and organic solvent is a water-miscible organic solvent or a mixture of such solvents. Preferred water-miscible organic solvents include C_{1-6}-alkanols, preferably methanol, ethanol, n-propanol, isopropanol, n-butanol, sec-butanol, tert-butanol, n-pentanol, cyclopentanol and cyclohexanol; linear amides, preferably dimethylformamide or dimethylacetamide; ketones and ketone-alcohols, preferably acetone, methyl ether ketone, cyclohexanone and diacetone alcohol; water-miscible ethers, preferably tetrahydrofuran and dioxane; diols, preferably diols having from 2 to 12 carbon atoms, for example pentane-1,5-diol, ethylene glycol, propylene glycol, butylene glycol, pentylene glycol, hexylene glycol and thiodiglycol and oligo- and poly-alkyleneglycols, preferably diethylene glycol, triethylene glycol, polyethylene glycol and polypropylene glycol; triols, preferably glycerol and 1,2,6-hexanetriol; mono-C_{1-4}-alkyl ethers of diols, preferably mono-C_{1-4}-alkyl ethers of diols having 2 to 12 carbon atoms, especially 2-methoxyethanol, 2-(2-methoxyethoxy)ethanol,

2-(2-ethoxyethoxy)-ethanol, 2-[2-(2-methoxyethoxy)ethoxy]ethanol, 2-[2-(2-ethoxyethoxy)-ethoxy]-ethanol and ethyleneglycol monoallylether; cyclic amides, preferably 2-pyrrolidone, N-methyl-2-pyrrolidone, N-ethyl-2-pyrrolidone, caprolactam and 1,3-dimethylimidazolidone; cyclic esters, preferably caprolactone; sulphoxides, preferably dimethyl sulphoxide and sulpholane. Preferably the liquid medium comprises water and 2 or more, especially from 2 to 8, water-soluble organic solvents.

Especially preferred water-soluble organic solvents are cyclic amides, especially 2-pyrrolidone, N-methyl-pyrrolidone and N-ethyl-pyrrolidone; diols, especially 1,5-pentane diol, ethyleneglycol, thiodiglycol, diethyleneglycol and triethyleneglycol; and mono- C_{1-4}-alkyl and C_{1-4}-alkyl ethers of diols, more preferably mono- C_{1-4}-alkyl ethers of diols having 2 to 12 carbon atoms, especially ((2-methoxy-2)-ethoxy)-2-ethoxyethanol.

Optionally the liquid medium comprises an oxidant.

Preferred low melting point solids have a melting point in the range from 60°C to 125°C. Suitable low melting point solids include long chain fatty acids or alcohols, preferably those with C_{18-24} chains, and sulphonamides. The compound of Formula (1) may be dissolved in the low melting point solid or may be finely dispersed in it.

Typically the liquid medium will further comprise one or more surfactants, for example anionic and/or nonionic surfactants. Examples of anionic surfactants include: Sulfonate surfactants such as Sulfosuccinates (Aerosol™ OT, A196; AY and GP, available from CYTEC) and Sulfonates (Aerosol™ DPOS-45, OS available from CYTEC; Witconate™ C-50H available from WITCO; Dowfax™ 8390 available from DOW); and Fluoro surfactants (Fluorad™ FC99C available from 3M). Examples of nonionic surfactants include: Fluoro surfactants (Fluorad™ FC170C available from 3M); Alkoxylate surfactants (Tergitol™ series 15S-5, 15S-7, and 15S-9 available from Union Carbide); and Organosilicone surfactants (Silwet™ L-77 and L-76-9 available from WITCO).

One or more buffers may optionally be included in the liquid medium to modulate pH of the ink. The buffers can be organic-based biological buffers or inorganic buffers, preferably, organic-based. Examples of preferably-employed buffers include tris(hydroxymethyl)aminomethane (TRIS), available from companies such as Aldrich Chemical (Milwaukee, Wis.), 4-morpholine — ethanesulfonic acid (MES), 4-morpholinepropanesulfonic acid (MOPS), and beta-hydroxy-4-morpholinepropanesulfonic acid (MOPSO). Further, the buffers employed should provide a pH ranging from about 3 to about 9 in the practice of the invention, preferably about 4 to about 6 and most preferably from about 4 to about 5.

One or more of the biocides commonly employed in inkjet inks may optionally be used in the ink, such as Nuosept™ 95, available from Huls America (Piscataway, N.J.); Proxel™ GXL, available from Zeneca (Wilmington, Del.); and glutaraldehyde, available from Union Carbide Company (Bound Brook, N.J.) under the trade designation Ucarcide 250.

Inks according to the invention may optionally also include one or more metal chelator. Such chelators are used to bind transition metal cations that may be present in the ink. Examples of preferred metal chelators include: ethylenediaminetetraacetic acid ("EDTA"); diethylenediaminepentaacetic acid ("DPTA"), trans-1,2-diaminocyclohexanetetraacetic acid ("CDTA"), ethylenedinitrilotetraacetic acid ("EGTA"), or other chelators.

In one embodiment inks according to the invention have a pH of from about 3 to about 6, preferably from about 3.5 to about 4.5. In another embodiment the pH of the composition is preferably from 4 to 11, more preferably from 7 to 10. Optionally the composition comprises a buffer.

The viscosity of the composition at 25°C is preferably less than 50cP, more preferably less than 20 cP and especially less than 5cP.

When the compositions according to the invention are used as ink jet printing compositions, the composition preferably has a concentration of less than 500 parts per million, more preferably less than 100 parts per million of halide ions. It is especially preferred that the composition has less than 100, more preferably less than 50 parts per million of divalent and trivalent metals, wherein parts refer to parts by weight relative to the total weight of the composition. We have found that purifying the compositions to reduce the concentration of these undesirable ions reduces nozzle blockage in ink jet printing heads, particularly in thermal ink jet printers. Similarly low levels as divalent and trivalent metals are also preferred.

The compounds of the invention may be used as the sole colorant in the compositions because of their attractive black shade. However, if desired, one may combine the present compounds together and/or with one or more further colorants to reduce nozzle blockage (by improving their solubility) or if a slightly different shade is required for a particular end use. The further colorants are preferably dyes. When further colorants are included in the composition these are preferably selected from black, magenta, cyan and yellow colorants and combinations thereof.

Suitable further black colorants include C.I.Food Black 2, C.I.Direct Black 19, C.I.Reactive Black 31, PRO-JET™ Fast Black 2, C.I.Direct Black 195; C.I.Direct Black 168; and black dyes described in patents by Lexmark (e.g. EP 0 539,178 A2, Example 1, 2, 3, 4 and 5), Orient Chemicals (e.g. EP 0 347 803 A2, pages 5-6, azo dyes 3, 4, 5, 6, 7, 8, 12, 13, 14, 15 and 16) and Seiko Epson Corporation.

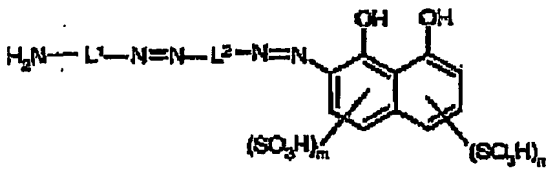
Suitable further magenta colorants include PRO-JET™ Fast Magenta 2 .

Suitable further yellow colorants include C.I.Direct Yellow 142; C.I.Direct Yellow 132; C.I.Direct Yellow 86; PRO-JET™ Yellow OAM; PRO-JET™ Fast Yellow 2; C.I.Direct Yellow 85; C.I. Direct Yellow 173; and C.I.Acid Yellow 23.

Suitable further cyan colorants include phthalocyanine colorants, C.I. Direct Blue 199 and C.I. Acid Blue 99.

The composition may also contain additional components conventionally used in ink jet printing inks, for example viscosity and surface tension modifiers, corrosion inhibitors, biocides, kogation reducing additives and surfactants which may be ionic or non-ionic.

In a fourth aspect of the invention we have also devised a process for the preparation of a compound of Formula (1) as hereinbefore defined which comprises diazotising an amine of Formula (3) and coupling the resultant diazonium salt with a compound of Formula A-H:



Formula (3).

The compound of Formula (3) may be prepared by diazotising a compound of Formula $CH_3CONH\text{-}L^1\text{-}N{=}N\text{-}L^2\text{-}NH_2$ and coupling the resultant diazonium salt onto a suitable 1,8-dihydroxy naphthalene compound, then removing the CH_3CO group by hydrolysis. The compound of Formula $CH_3CONH\text{-}L^1\text{-}N{=}N\text{-}L^2\text{-}NH_2$ may be prepared by diazotising an amine of formula $CH_3CONH\text{-}L^1\text{-}NH_2$ and coupling onto an amine of Formula $H\text{-}L^2\text{-}NH_2$.

Preferably the diazotisations are carried out using a diazotising agent, especially sodium nitrite under acidic conditions. Further preferably the diazotisations are carried out at a temperature of 0 to 5°C. In the above process A, L^1, L^2, m and n are as hereinbefore defined.

A further aspect of the present invention provides a paper, an overhead projector slide or a textile material printed with a composition, a compound or by means of a process according to the present invention.

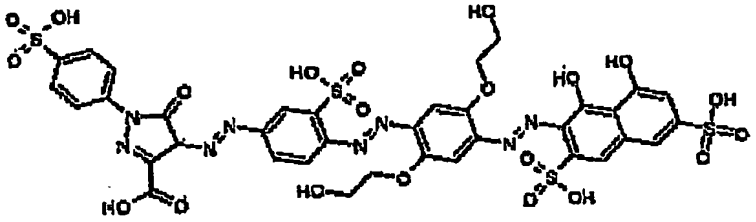
A still further as aspect of the present invention provides an ink jet printer cartridge, optionally refillable, comprising one or more chambers and a composition, wherein the composition is present in at least one of the chambers and the composition is as defined in the third aspect of the present invention.

The present compounds and compositions provide prints of attractive, neutral black shades that are particularly well suited for the ink jet printing of text and images. The compositions have good storage stability and low tendency to block the very fine nozzles used in ink jet printers. Furthermore, the resultant images have good optical density, light-fastness, wet-fastness and resistance to fading in the presence of oxidising air pollutants (e.g. ozone).

The invention is further illustrated by the following Examples in which all parts and percentages are by weight unless specified otherwise. The abbreviation "Ac" means CH_3CO- .

Example 1

Preparation of:



Preparation of intermediate 2,5-di-(2-acetoxyethoxy)aniline

Step 1 - Preparation of 1,4-bis-(2-acetoxyethoxy)hydroquinone

Hydroquinonebis-(2-hydroxyethyl)ether (179g), acetic acid (100ml) and acetic anhydride (300ml) were stirred and heated under reflux overnight. After cooling to room temperature and drowning into water (2l) the product was isolated by filtration, washed with water, dried and recrystallised from ethanol to give 212g of product.

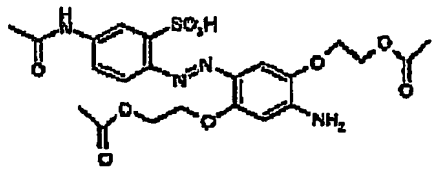
Step 2 - Preparation of 2-Nitro-1,4-bis-(2-acetoxyethoxy)hydroquinone

The product of step 1 (211.5g) was dissolved in acetic acid (1800ml). A mixture of nitric acid (51.9ml) and acetic acid (200ml) was then added over 20 minutes keeping the temperature below 20°C. After stirring at room temperature overnight the solution was drowned into water (9l) and the product isolated by filtration, washed with water and recrystallised from ethanol to give 209g of product.

Step 3 - Preparation of 2,5-di-(2-acetoxyethoxy)aniline

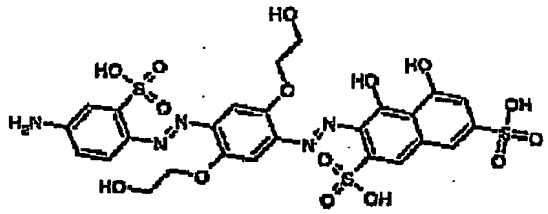
2-Nitro-1,4-bis-(2-acetoxyethoxy)hydroquinone (115g) was dissolved in ethanol at 50°C and reduced with hydrogen in the presence of palladium catalyst (2g, 5%Pd/C). When uptake of hydrogen ceased the solution was screened to remove the catalyst and the filtrates allowed to cool to room temperature. The crystalline solid was isolated by filtration and dried under vacuum to give 90g of product.

Stage one - Preparation of monoazo-4-(4-Acetylamino-2-sulpho-3-phenylazo)-2,5-di-(2-acetoxyethoxy)aniline



4-Amino-3-sulphoacetanilide (174g; 0.6 mol) was stirred in water (2.5l) at pH 9 and sodium nitrite (45.54g; 0.66 mol) added. The solution was added to ice/water containing concentrated hydrochloric acid (180ml) with stirring. After stirring for 1.5h at less than 10°C the excess nitrous acid was destroyed by the addition of sulphamic acid. 2,5-di-(2-acetoxyethoxy)aniline (178.2g; 0.6mol) was dissolved in acetone (1000 ml) and added to the above diazonium salt suspension at 0-10°C followed by the slow addition of pyridine (30ml). After stirring overnight at room temperature the precipitated product was filtered-off, washed with water. The damp paste was then stirred in acetone; filtered and dried (50°C) to give a an orange solid (210g; 64%).

Stage two – Preparation of bisazo intermediate



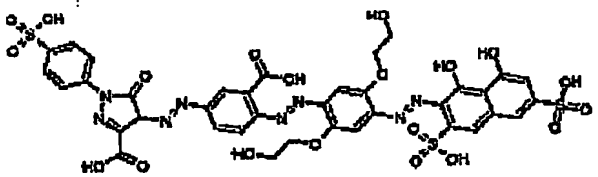
The monoazo product from Stage one (24.75g; 0.05mol) was dissolved in water (300ml) with stirring at pH 10 to which sodium nitrite (6.90g; 0.1mol) and acetone (200ml) were added. The resulting mixture was then added to 0.10M hydrochloric acid (70ml) with stirring at room temperature. After stirring for 1h, the excess nitrous acid was destroyed by the addition of sulphamic acid. The resulting diazonium salt was then added to a stirred solution of chromotropic acid (20.00g; 0.05mol) at 0 - 10°C at pH 7- 8 maintained by the addition of 2N lithium hydroxide when necessary. After stirring overnight the product was precipitated by the addition of 25% (w/v) lithium chloride then filtered and washed with 30% (w/v) lithium chloride solution. The resulting damp paste was suspended in water (700ml) and lithium hydroxide hydrate (25.00g; 0.60mol) added and the solution heated at 70°C. After 3h the solution neutralised to pH 6 – 7 by the addition of concentrated hydrochloric acid. The product was precipitated by the slow addition of 20 % (w/v) lithium chloride, filtered and washed with 25% (w/v) lithium chloride solution. The damp paste was dissolved in water and then dialysed to low conductivity. The solution was evaporated to dryness (70 °C) to give a black powder (25.5g; 67%)

Stage Three- preparation of title dye

The amino disazo compound from Stage Two (12.00g; 0.0158mol) was dissolved in water (250ml) with stirring at pH 9 to which calsolene oil (1ml) and sodium nitrite (1.20g; 0.0174 mol) was added. The resulting solution was then added to ice / water (100g) containing concentrated hydrochloric acid (5ml) with stirring at 0 -10°C . After stirring for 1h at 0 -10°C the excess nitrous acid was destroyed by the addition of sulphamic acid. The resulting diazonium salt was added to a stirred solution of 1-(4-sulphophenyl)-3-carboxy-5-pyrazolone (5.39g; 0.19mol) in water (100ml) at 0 - 10°C and then adjusted to pH 7. After stirring overnight the solution was poured into acetone (3l) with stirring, filtered and washed with acetone. The solid dissolved in water and dialysed to low conductivity to give after evaporation (80°C) a black powder (11.61g; 68.8%; λ_{max} 612nm and an ε_{max} of 82232; mass spectrum (M-H)-ve 1037).

Example 2

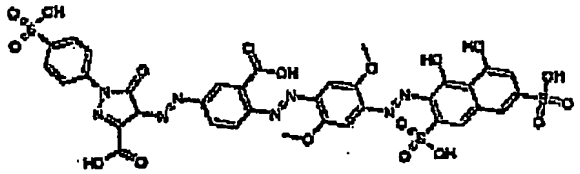
Preparation of:



The method of Example 1 was repeated except that in place of 4-amino-3-sulpho acetanilide there was used 4-amino-3-carboxyacetanilide. The resultant compound had a λ_{max} at 602nm and an ε_{max} of 79,227.

Example 3

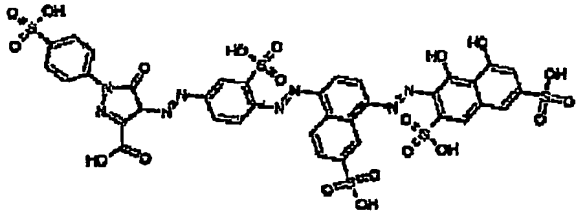
Preparation of:



The method of Example 2 was repeated except that in place of 2,5-di-(2-acetoxyethoxy)aniline there was used 2,5-di-(methoxy)aniline. The resultant compound had a λ_{max} at 601nm.

Example 4

Preparation of:

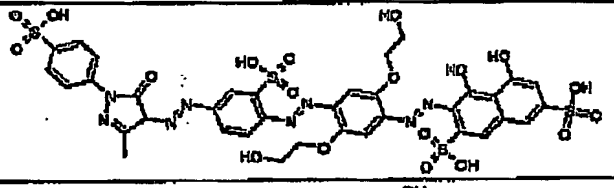
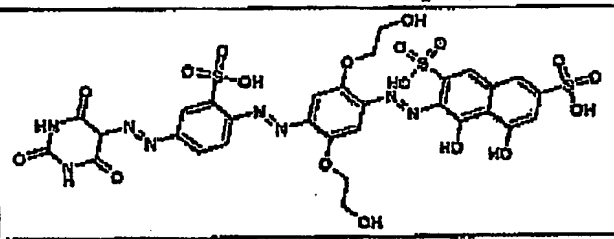
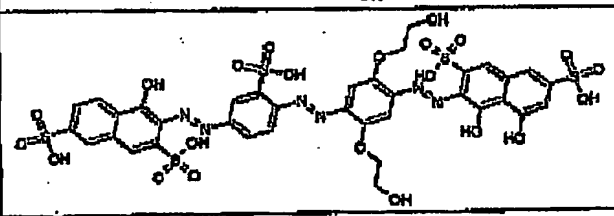
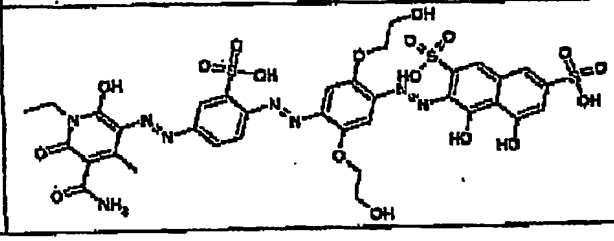


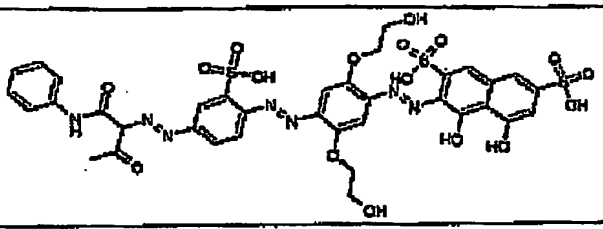
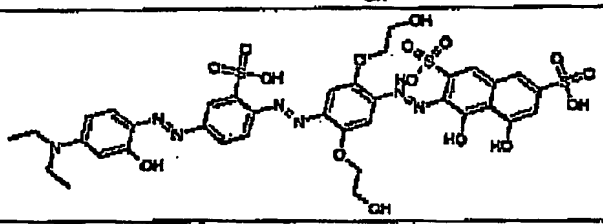
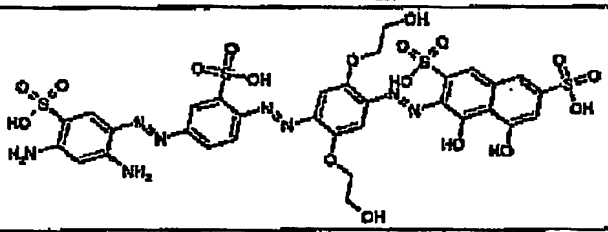
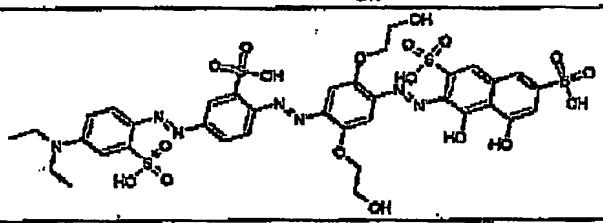
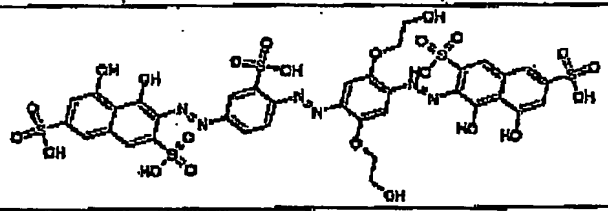
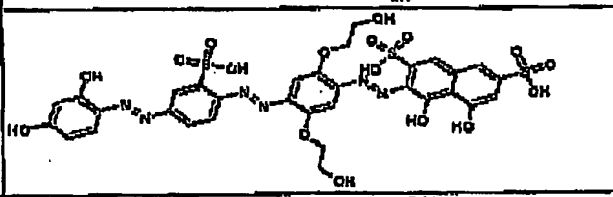
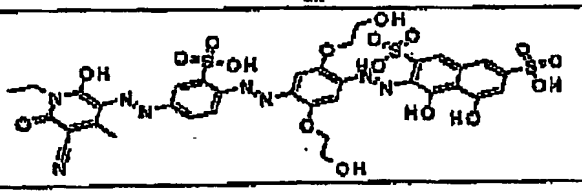
The method of Example 1 was repeated except that in place of 2,5-di-(2-acetoxyethoxy)aniline there was used 1-amino-7-sulpho naphthalene. The resultant compound had a λ_{max} at 604 nm and an ε_{max} of 69,419.

Examples 5 to 15

Examples 5 to 15 shown in Table 1 were prepared by following the general method of Example 1, except that in place of 1-(4-sulphophenyl)-3-carboxy-5-pyrazolone there was used the compound described in Column A of Table 1.

Table 1

Ex	A	Compound	ε_{max}	λ_{max}(nm)
5	1-(4-sulphophenyl)-3-methyl-5-pyrazolone		72926	604
6	Barbituric acid		73364	604
7	1-Hydroxy-3,6-disulpho naphthalene		87995	618
8	3-Carbamoyl-4-methyl-6-hydroxy-N-ethyl pyridone		81232	601

9	Acetoacetanilide		61463	611
10	3-(N,N-diethylamino) phenol		82378	612
11	3-amino-4-sulpho aniline		81414	609
12	3-Diethylamino benzenesulphonic acid		81685	600
13	1,8-Dihydroxy-3,6-disulpho naphthalene (Chromotropic acid)		99865	626
14	Resorcinol		85064	612
15	1-ethyl-1,2-dihydro-6- hydroxy-4-methyl-2-oxo-3-pyridinecarbonitrile		75,785	610

Examples 16 to 25 – Mixtures

The following mixtures described in Table 2 may be prepared in which the bracketed number is the number of parts by weight of the relevant compound:

Table 2

Example	Compound (parts)	Compound (parts)
16	Example 8 (1)	Example 1 (0.9)
17	Example 4 (1)	C.I. Direct Yellow 132 (0.1)
18	Example 8 (1)	Example 13 (0.5)
19	Example 15 (1)	Example 14 (1)
20	Example 8 (1)	C.I. Direct Blue 199 (0.15)
21	Example 1 (1)	Example 8 (1)
22	Example 8 (1)	Pro-Jet™ Fast Black 2 (0.7)
23	Example 12 (1)	Example 14 (0.5)
24	Example 8 (1)	C.I. Direct Yellow 86 (0.12)
25	Example 3 (1)	Example 8 (0.5)
26	Example 8 (1)	Pro-Jet™ Yellow OAM (0.05)
27	Example 1 (1)	Example 4 (1)
28	Example 4 (1)	Pro-Jet™ Fast Yellow 2 (0.05)
29	Example 1 (1)	Pro-Jet™ Fast Magenta 2 (0.05)

Example 30 - Ink Formulations

Inks may be prepared according to the following formulation wherein Dye is the compound or mixture from each of the above Examples above:

2-Pyrrolidone 5 parts
Thiodiglycol 5 parts
Surfynol™ 465 1 part (from Air Products Inc., USA)
Dye 3 parts
Water 86 parts

Further inks described in Tables 3 and 4 may be prepared wherein the Dye described in the first column is the compound or mixture made in the above Example of the same number. Numbers quoted in the second column onwards refer to the number of parts of the relevant ingredient and all parts are by weight. The inks may be applied to paper by thermal or piezo ink jet printing.

The following abbreviations are used in Table 3 and 4:
PG = propylene glycol
DEG = diethylene glycol

NMP = N-methyl pyrollidone
DMK = dimethylketone
IPA = isopropanol
MEOH = methanol
2P = 2-pyrollidone
MIBK = methylisobutyl ketone
P12 = propane-1,2-diol
BDL = butane-2,3-diol
CET= Tris(2-aminoethyl)amine buffer
PHO = Na_2HPO_4 and
TBT = tertiary butanol
TDG = thiodiglycol

TABLE 3

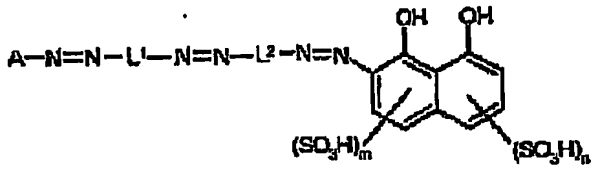
Dye:	Dye Content	Water	PG	DEG	NMP	DMK	NaOH	Na Stearate	IPA	MEOH	2P	MIBK
1	2.0	80	5		6	4					5	
2	3.0	90		5	5		0.2					
3	10.0	85	3		3	3				5	1	
4	2.1	91		8								1
5	3.1	86	5					0.2	4			5
6	1.1	81			9		0.5	0.5			9	
7	2.5	60	4	15	3	3			6	10	5	4
8	5	65		20					10			
9	2.4	75	5	4		5				6		5
10	4.1	80	3	5	2	10		0.3				
11	3.2	65		5	4	6			5	4	6	5
12	5.1	96								4		
13	10.8	90	5						5			
14	10.0	80	2	6	2	5			1		4	
15	1.8	80		5							15	
16	2.6	84			11						5	
17	3.3	80	2			10				2		6
18	12.0	90				7	0.3		3			
19	5.4	69	2	20	2	1					3	3
20	6.0	91			4						5	

TABLE 4

Dye	Dye Content	Water	PG	DEG	NMP	CET	TBT	TDG	BDL	PHO	2P	PI2
21	3.0	80	15			0.2					5	
22	9.0	90		5						1.2		5
23	1.5	85	5	5		0.15	5.0	0.2				
24	2.5	90		6	4					0.12		
25	3.1	82	4	8		0.3						6
8	0.9	85		10					5	0.2		
8	8.0	90		5	5			0.3				
8	4.0	70		10	4				1		4	11
8	2.2	75	4	10	3				2		6	
8	10.0	91			6						3	
8	9.0	76		9	7		3.0			0.95	5	
8	5.0	78	5	11							6	
8	5.4	86			7						7	
8	2.1	70	5	5	5	0.1	0.2	0.1	5	0.1	5	
8	2.0	90		10								
8	2	88						10				
8	5	78			5			12			5	
8	8	70	2		8			15			5	
8	10	80						8			12	
8	10	80		10								

Claims

1. A process for printing an image on a substrate comprising applying thereto a composition comprising a liquid medium and a tris-azo compound of Formula (1) or salt thereof:



Formula (1)

wherein:

A is an optionally substituted alkenyl, homocyclic or heterocyclic group;

L^1 and L^2 are each independently optionally substituted aryl or heteroaryl; and

m and n are each independently 0 or 1 such that m+n is 1 or 2;

wherein:

(i) the compound of Formula (1) is optionally in the form of a metal chelate; and

(ii) at least one of L^1 and L^2 carries at least one substituent selected from sulpho, carboxy, C_{1-4}-alkoxy and C_{1-4}-alkoxy-OH.

2. A process according to claim 1 wherein the composition is applied to the substrate by means of an ink jet printer.

3. A process according to any one of the preceding claims wherein the image is text, a picture, a photorealistic image or a combination thereof.

4. A process according to any one of the preceding claims wherein the substrate is paper, plastic, metal or glass.

5. A process according to any one of the preceding claims wherein:

A is optionally substituted pyridyl, furyl, thienyl, thiazolyl, isothiazolyl, imidazolyl, benzimidazolyl, pyrazinyl, pyrimidyl, quinolyl, isoquinolyl, benzofuryl, benzothienyl, pyrazolyl, indolyl, purinyl, isoxazolyl, oxazolyl, thiadiazolyl, furazanyl, pyridonyl, pyrazolonyl, piperidinyl, piperazinyl, pyrrolidinyl, morpholinyl, tetrahydrofuranyl, tetrahydrothiophenyl or tetrahydropyranyl;

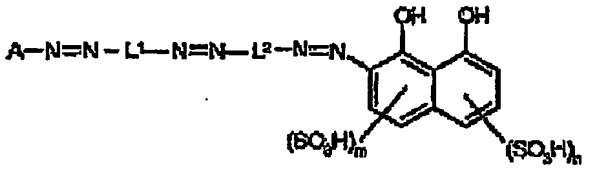
L^1 phenyl or naphthyl optionally carrying a substituent selected from sulpho and carboxy;

L^2 is phenyl or naphthyl carrying at least one substituent selected from sulpho, carboxy C_{1-4}-alkoxy and C_{1-4}-alkoxy-OH; and

m and n are each independently 0 or 1 such that m+n is 1 or 2;

wherein said optional substituents are selected from OH; SO_3H; CN; carbonamido; PO_3H_2; CO_2H; NO_2; NH_2; C_{1-4}-alkyl optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group; C_{1-4}-alkoxy optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, C_{1-4}-alkyl, amino or hydroxy group; phenyl or phenyl carrying from 1 to 3 substiuents selected from sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino, hydroxy and N carrying one or two C_{1-4}-alkyl groups optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group; N carrying one or two C_{1-4}-alkyl groups optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group; and C_{1-4}-acylamino.

6. A tris-azo compound of Formula (1) or salt thereof:



Formula (1)

wherein:

A is an optionally substituted alkenyl, homocyclic or heterocyclic group;

L^1 and L^2 are each independently optionally substituted aryl or heteroaryl;

m and n are each independently 0 or 1 such that m+n is 1 or 2; and

with the provisos that (i) the compound of Formula (1) is optionally in the form of a metal chelate; (ii) at least one of L1 and L2 carries at least one substituent selected from sulpho, carboxy, C1-4-alkoxy and C1-4-alkoxy-OH; and (iii) when L^1 carries a methoxy group A is not 1,3-diaminophenyl.

7. A compound according to claim 6 wherein A is optionally substituted pyridyl, furyl, thienyl, thiazolyl, isothiazolyl, imidazolyl, benzimidazolyl, pyrazinyl, pyrimidyl, quinolyl, isoquinolyl, benzofuryl, benzothienyl, pyrazolyl, indolyl, purinyl, isoxazolyl, oxazolyl, thiadiazolyl, furazanyl, pyridonyl, pyrazolonyl, piperidinyl, piperazinyl, pyrrolidinyl, morpholinyl, tetrahydrofuranyl, tetrahydrothiophenyl or tetrahydropyranyl.

8. A compound according to claim 6 wherein A is optionally substituted pyridonyl.

9. A compound according to any one of claims 6 to 8 wherein the optional substituents are selected from OH; SO_3H; CN; carbonamido; PO_3H_2; CO_2H; NO_2; NH_2; C_{1-4}-alkyl optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group; C_{1-4}-alkoxy optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, C_{1-4}-alkyl, amino or hydroxy group; phenyl or phenyl carrying from 1 to 3 substituents selected from sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino, hydroxy and N carrying one or two C_{1-4}-alkyl groups optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group; N carrying one or two C_{1-4}-alkyl groups optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group; and C_{1-4}-acylamino.

10. A compound according to any one of claims 6 to 9 wherein L^1 is phenyl or naphthyl optionally carrying a substituent selected from sulpho and carboxy.

11. A compound according to any one of claims 6 to 10 wherein L^2 is phenyl or naphthyl carrying at least one substituent selected from sulpho, carboxy C_{1-4}-alkoxy and C_{1-4}-alkoxy-OH.

12. A compound according to any one of claims 6 to 11 wherein L^2 is phenyl or carrying two substituents selected from C_{1-4}-alkoxy-OH.

13. A compound according to claim 6 wherein:

A is optionally substituted pyridyl, furyl, thienyl, thiazolyl, isothiazolyl, imidazolyl, benzimidazolyl, pyrazinyl, pyrimidyl, quinolyl, isoquinolyl, benzofuryl, benzothienyl, pyrazolyl, indolyl, purinyl, isoxazolyl, oxazolyl, thiadiazolyl, furazanyl, pyridonyl, pyrazolonyl, piperidinyl, piperazinyl, pyrrolidinyl, morpholinyl, tetrahydrofuranyl, tetrahydrothiophenyl or tetrahydropyranyl;

L^1 phenyl or naphthyl optionally carrying a substituent selected from sulpho and carboxy;

L^2 is phenyl or naphthyl carrying at least one substituent selected from sulpho, carboxy C_{1-4}-alkoxy and C_{1-4}-alkoxy-OH; and

m and n are each independently 0 or 1 such that m+n is 1 or 2;

wherein said optional substituents are selected from OH; SO_3H; CN; carbonamido; PO_3H_2; CO_2H; NO_2; NH_2; C_{1-4}-alkyl optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group; C_{1-4}-alkoxy optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, C_{1-4}-alkyl, amino or hydroxy group; phenyl or phenyl carrying from 1 to 3 substituents selected from sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino, hydroxy and N carrying one or two C_{1-4}-alkyl groups optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group; N carrying one or two C_{1-4}-alkyl groups

optionally carrying a sulpho, carboxy, phosphato, C_{1-4}-alkoxy, amino or hydroxy group; and C_{1-4}-acylamino.

14. A compound as defined in any one of the Examples described herein.

15. A composition comprising a compound of Formula (1) or salt thereof as defined in claim 1 and a low melting point solid or a liquid medium comprising water and an organic solvent.

16. A composition according to claim 15 wherein the compound of Formula (1) is as defined in any one of claims 6 to 14.

17. A composition according to claim 15 or 16 which has a concentration of less than 500 parts per million of halide ions, wherein parts refer to parts by weight relative to the total weight of the composition.

18. A composition according to claim any one of claims 15 to 17 which has less than 50 parts per million of divalent and trivalent metals, wherein parts refer to parts by weight relative to the total weight of the composition.

19. A paper, an overhead projector slide or a textile material printed with a composition according to claim 15, 16, 17 or 18 or a compound according to any one of claims 6 to 14 or by means of a process according to any one of claims 1 to 5.

20. An ink jet printer cartridge, optionally refillable, comprising one or more chambers and a composition, wherein the composition is present in at least one of the chambers and the composition is as defined in any one of claims 15 to 18.

21. Use of a compound of Formula (1), as defined in claim 1, in ink jet printing.